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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: AN ORGANIC MINERAL FERTILIZER

(57) Abstract: The invention relates to the preparation method of an organic mineral fertilizer from organic material using dry solid granulation technique with urea. Further, the invention relates to the fertilizer product having good mechanical properties. The method of the invention for preparing organic waste material based fertilizer granules comprises the steps of: drying the organic waste material and crushing the dried waste, mixing the crushed waste material with dry urea and optionally with other fertilizer source materials to provide a solid feed material, feeding the said solid feed to a melter, heating the feed material for melting a desired portion thereof and keeping said portion in a molten state, feeding the partly molten feed material from the melter to a granulator or to obtain a granulated product, cooling and screening the granulated product to obtain dry and mechanically resistant organic mineral fertilizer granules having a desired size distribution.

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An organic mineral fertilizer

The invention relates to the preparation method of an organic mineral fertilizer from organic material using dry solid granulation technique with urea. Further, the invention relates to the fertilizer product having good mechanical properties.

Large amounts of natural wastes are produced through the usage of nitrogenous natural materials and they present a disposal problem. Most of this waste material is available in a form generally commercially unusable in agriculture. This waste material includes waste and byproducts from life stock production, breeding and processing, waste from the kept animals to recovered sludges from sewage treatment plants.

A strong interest has focused on the use of these natural nitrogenous waste materials for fertilizers in agriculture. The typical fertilizer products have been results of composting, extrusion, compacting, pelletizing or inclusion in resinous agglomerated together with a few methods of conversion into granules.

Harmony Products Inc. has disclosed in US 5,021,077 a method of preparing granules for agriculture by heating moist natural nitrogenous materials such as grain by-products, hair, feather, seafood, blood and bone meals and poultry waste, under alkaline conditions. The organic material was mixed with aldehyde compounds in a fluid bed while the materials exhibited adhesive properties. Subsequently, the material was granulated and hardened by drying it in the air with further heating.

Similarly, they have disclosed in US 5,411,568 a method for preparing a slow release fertilizer by mixing dry conditioned nitrogenous particulate waste with ureaformaldehyde oligomer. First, the organic nitrogenous waste was dried and screened, and a reactive ureaformaldehyde liquid oligomer from urea and formaldehyde was prepared. The waste and oligomer were mixed until the oligomer was evenly distribute in the waste granules, and subsequently, mineral acid was added for regulating the pH and adjusting pH further for obtaining a slow release product. The crushing test for larger granules between 8 and 6 mm indicated fairly hard particles of an average crushing force of 13 N.

One issue for a farmer using the organic material based fertilizers is the mechanical resistance and robustness of the granules. They should be hard enough to withstand machine spreading which is conventionally used for mineral fertilizers. The

crushing strength required and typically achieved with mineral fertilizers is about 30-40 N.

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The solid granulation technique as described in our previous patent application WO-A 00/00452 was developed to solve the granulation, product quality and storage problems in manufacturing compound fertilizers. It is a process for the preparation of compound fertilizers such as N-P-K, N-K or N-P where solid raw materials are mixed in a mixer and fed to a granulator together with hot air. The raw materials are granulated without the addition of water or any other liquid such as ammonia, phosphoric acid or sulphuric acid. Thus the granulation is a true solid granulation process. Because neither water nor any other liquid is added there is no need for drying the granulated product. Furthermore, the physical quality of the product is good. The process of this technique was further developed as described in our patent FI-A 19992021.

It is an object of this invention to process organic waste into granulated fertilizer form for it to be industrially usable. Typically, for example composted material when used as such can satisfy only local needs, it is seldom transported very far.

It is a further object of this invention to achieve an organic waste material based fertilizer with good mechanical properties to be used similarly to, for example, a mineral based field fertilizer.

A new method has been discovered for preparing organic waste based high integrity fertilizer granules. It was found that prior to mixing dried organic waste material can be processed into mechanically durable granules by using the solid granulation technique. By heating the essentially water-free mixture of dried organic waste material together with urea and with optionally other fertilizer source materials mechanically durable fertilizer granules comparable with a field fertilizer nutrition values could be obtained.

The new method of the present invention for preparing organic waste material based fertilizer granules comprises the steps of:

- a) drying the organic waste material and optionally crushing the dried waste,
- b) mixing the dried organic waste material with dry urea and optionally with other fertilizer source materials and recycle material to provide a solid feed material having a desired composition,

- c) feeding the said solid feed to a melter and introducing heating for the feed material and for melting a desired portion thereof and keeping said portion in a molten state,
- d) feeding the partly molten feed material from the melter to a granulator to obtain a granulated product,
- e) cooling and screening the granulated product to obtain dry and mechanically resistant organic mineral fertilizer granules having a desired size distribution.

According to a preferred embodiment of the invention, the process is carried out continuously and the molten portion of the feed material is kept constant during the process by controlling the flow rate of the feed material and the temperature of the heater media heating up the melter. The optimal portion of the molten feed material depend on the grade of the fertilizer wanted and the source materials used.

The melter is heated either externally or internally. The needed temperature is dependent on the composition of the fertilizer feed. The melter walls can be heated externally using electrical heater elements. Preferably, hot dry air is directed to the melter the temperature of which is between 300 °C and 550 °C. As the air leaves the melter the temperature has typically decreased down to below 105 °C. The temperature of the feed material to be fed to the melter from the mixer is between 30 °C to 60 °C.

Suitably, the temperature of the partly molten feed material leaving the melter is between 70 °C and 110 °C.

The organic waste materials which can be used in the present invention are for example composted animal excrete, swine, cattle or poultry manure, composted slaughterhouse waste, recovered sludge from sewage treatment plants, grain by-products or peat, preferably swine or cattle manure. The organic waste material is dried prior to further use to a water content of less than 10%. Optionally, the material is crushed to a particle size of less than two or one millimetres depending on the desired product size.

The dry urea used according to the invention can be of different grades, for example, powdery or in form of prills. The content of urea to be mixed with the organic waste material is at least 40%. If no other fertilizer start material is used, the content of urea may be up to 80%.

In the preferred embodiment of the method, other solid fertilizer source materials can be used for adjusting the fertilizer nutrition value and the final composition of the fertilizer material. For example, diammonium phosphate (DAP), monoammonium phosphate (MAP), phosphate rock, potassium chloride (MOP), potassium sulphate (SOP), single superphosphate (TSP), ammonium sulphate (AS) and ammonium chloride could be used.

Before entering commercial suppliers, the granulated product is screened to obtain fertilizer granules of a size between 2 mm and 5 mm. Granule size may vary depending on the customer needs. The amount of offsize screen is small, less than 2 % for the granules over 5 mm and the same for granules below 2 mm. The undersize material (< 2 mm) and the oversize material (>5 mm) obtained in the screening can be recirculated as said recycle material. Optionally, the oversize material can be milled after the screening before being recirculated.

The melter and the granulator can be separate units but the melter and the granulator can also be part of the same equipment.

The final granulated product has a low water content, depending on the grade less than two or one weight percent, originating from the source materials. This water content is low enough, there is no need for drying the product further as in the methods where water or other liquid is added to the fertilizer mixture before or during granulation. In the method according to the invention water is vaporized due to the release of generated crystallization heat from the urea.

By the method according to the invention granules of high integrity and good mechanical properties are obtained. The crushing force of the granules is at least 25 N, which is of high importance considering the large scale use of the granules in agriculture by means of machine spreading of the granules. Abrasion and the shattering resistances of the product are very good, less than 0.5% and less than 30%, respectively.

A variety of fertilizer grades can be produced according to the method of this invention. The grade depends on the source materials used. When using only urea and composted manure, N-type fertilizer is produced. Depending on the quantity of the other fertilizer source materials and the quality of the organic waste material, N-K or N-P-K fertilizers of varying grades are obtained.

This invention has advantages over other organic waste based fertilizer granulation methods in that materials can be granulated without the use of additional moisture

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or water or other liquids such as mineral acids, for example. Thus, there is no need for an expensive granule drying step. In addition granules made by this invention are mechanically of good quality enabling the use of a large scale machine spreading.

The following examples will illustrate the invention in more detail.

Example 1

Organic waste material consisting of composted swine and cattle manure comprising the chemical composition listed in Table 1 was dried to a water content of 5.6% determined by the Karl-Fisher method. The dried, composted material was crushed to a grain size below 1 mm using Atrex mill.

A mixture was made from 52% urea, 18,2% MAP (Russia, crushed), 15,8% KCl (K₂O 60%) and 14% dried and crushed composted material.

The mixture was fed into a bench scale blunger at the rate of 3.18 kg/h and it was recycled at the rate of 0.8 kg/h. The mixture was held at 97 °C by the introduction of heated dry air of 460 °C. Melting and granulating were carried out mainly at a granulation drum and partly at the cooler. The inside temperature of the cooler was 31 °C and the granules leaving the cooler were 26 °C.

Table 1. The composition of the composted source material

Water	27.8 %
Dry matter	71.7 %
Total carbon	21.4 %
Total nitrogen	2.6 %
NH4-N	0.5 %
Total P ₂ O ₅	4.2 %
Total K ₂ O	3.9 %

The fertilizer product thus obtained was coated with 2 kg/ton of SK Fert FW5 and 3 kg/ton of talc.

Example 2

The chemical and physical properties of the fertilizer material prepared according to Example 1 were determined.

Table 2 shows that the amount of soluble phosphor was about 60% and that the product has a N-P-K ratio of 23-8-10 which is suitable for a field fertilizer.

Table 2. The chemical analysis of the fertilizer product

0.34 %
17.4 %
23.4 %
21.6 %
1.7 %
8.2 %
7.9 %
4.9 %
10.6
5.6

29% of the product grain size was between 5-2.5 mm after the cooler, which indicates reasonable granulation efficiency. The sieve analysis (Table 3) of the product showed that 99% of the material was between 5 mm to 2 mm grain size. Caking in u-bags did not occur and the crushing strength was good. These features fulfil the requirement for using a field spreading machine which is essential for a farmer.

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Table 3. Physical properties of the fertilizer granules

Sieve analysis (%)	
+5	0
-5+2	99
-2	1
Caking in u-bags	0
Granule crushing strength (N)	27
Abrasion (%)	0.4
Shattering resistance (%)	28
CRH (%)	16
Moisture adsorption 80% RH	
2 h (%)	3.8
4 h (%)	6.9
6 h (%)	9.6

Example 3

Fertilizer granules were prepared as in example 1 scale, but with a composition of 48% urea, 16.9% MAP (Russia, crushed), 14.7% KCl (K₂O60 %) and 20% dried and crushed composted material.

Granulation was good in this case too, but the size distribution using the sieve from 5 to 2.5 mm decreased to about 18% indicating a lower capacity than in the Example 2.

We claim

1. A method for the preparation of high integrity organic mineral fertilizer granules from organic source material by solid granulation, said method comprising the steps of:

- a) drying and optionally crushing the organic source material,
- b) mixing the said organic source material with dry urea and optionally with other fertilizer source materials to provide a solid feed material having a desired composition,
- c) feeding the said solid feed to a melter and heating the feed material in a manner that a desired portion thereof melts and keeping said portion in a molten state,
- d) feeding the partly molten feed material from the melter to a granulator to obtain granulated product, and
- e) cooling and screening the granulated product to obtain dry and mechanically resistant organic mineral fertilizer granules having a desired size distribution.
- 2. A method according to claim 1, characterized in that the method is carried out as a continuous process.
- 3. A method according to claim 1, characterized in that the portion of organic source material in the solid feed is less than 30%.
- 4. A method according to claim 1, characterized in that the water content of the organic source material after drying is less than 10 %.
- 5. A method according to claim 1, characterized in that the content of urea is at least 40%.
- 6. A method according to any of claims 1-5, characterized in that the feed material is heated to the temperature between 70 °C and 110 °C and kept at that temperature to ensure that the feed material stays in partly molten state.
- 7. A method according to any of claims 1-7, characterized in that the other fertilizer source materials are selected from the group of monoammonium phosphate (MAP), diammonium phosphate (DAP), potassium sulphate (SOP), potassium

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chloride (MOP), phosphate rock, single superphosphate (SSP), triple superphosphate (TSP) and ammonium sulphate (AS).

- 8. A method according to claim 1, characterized in that the organic source material can comprise composted animal excrete, peat, waste from life stock processing.
- 9. A method according to claim 1, characterized in that the organic source material comprises preferably composted cow and pig manure.
- 10. A method according to claim 7 or 8, characterized in that the fertilizer can comprise at least one of the said source materials.
- 11. A method according to claim 1, characterized in that the undersize and the oversize material obtained in the screening are recirculated and that oversize material is optionally being milled after screening.

International application No.

PCT/FI 01/00667

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C05G 1/00, C05G 5/00, C05C 9/00, C05F 3/00, C05F 11/02, B01J 2/00 According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: C05C, C05F, C05G, B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2112765 A (MELAMINE CHEMICALS INC), 27 July 1983 (27.07.83), page 1, line 1 - line 23; page 2, line 1 - page 3, line 65; page 5, line 32 - line 46, page 8, line 40 - line 62, abstract	1-11
	ELL UDY D	1.11
X	File WPI, Derwent accession no.1995-252194, Denki Kagaku Kogyo KK: "Prodn. of a granular calcium cyanamide fertiliser, suitable for storage- by opt. including phosphate salt and organic fertiliser then granulating using urea", JP,A,157385, 19950620, DW199533	1-11
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LXI	Further documents are listed in the continuation of Box	C.	X See patent family annex.
*	Special categories of cited documents:	"T"	later document published after the international filing date or priority
"A"	document defining the general state of the art which is not considered to be of particular relevance		date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E"	earlier application or patent but published on or after the international filing date	"X"	document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive
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"O"	document referring to an oral disclosure, use, exhibition or other means		combined with one or more other such documents, such combination being obvious to a person skilled in the art
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C (Continu	ation). DOCUMENTS CONSIDERED TO BE RELEVANT	
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	WO 0000452 A1 (KEMIRA AGRO OY), 6 January 2000 (06.01.00), page 4, line 4 - page 6, line 32, claims 1-18, abstract	1-11
A	File WPI, Derwent accession no. 1982-54174E, As Belo Gen Inorg et al: "Prodn. of granulated urea-based fertiliser - includes addn. of powdered enriching seaweed, to urea melt before granulation", SU,B,859339, 19810901, DW198226	1-11
A	File WPI, Derwent accession no. 2000-497583, Malanchuk V Ya et al: "Organo-mineral fertilizer production process", RU,C1,2140408, 19991027, DW200044	7,8
A	GB 1603645 A (ENCIK LEET KENG CHOON), 25 November 1981 (25.11.81), page 2, line 20 - line 37, claim 1	7,9
A	US 5021077 A (MOORE), 4 June 1991 (04.06.91), column 1, line 1 - line 41, abstract	8,9
A	US 5411568 A (MOORE), 2 May 1995 (02.05.95), page 1, line 1 - line 24; page 4, line 17 - line 28, abstract	8
P,A	WO 0121556 A1 (KEMIRA AGRO OY), 29 March 2001 (29.03.01), claims 1-14, abstract	1-11
	; —— · · · · · · · · · · · · · · · · · ·	

Information on patent family members

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International application No.
PCT/FI 01/00667

	document arch report		Publication date	Patent family member(s)		Publication date
10	0000452	A1	06/01/00	AU	4912399 A	17/01/00
				BR	9911721 A	20/03/01
				CN	1307550 T	08/08/01
				EP	1127035 A	29/08/01
				FI	105807 B	00/00/00
				FI	981490 A	30/12/99
				FI	982013 A	30/12/99
				NO	20006487 A	19/12/00
₿B	1603645	A	25/11/81	NONE		
JS	5021077	Α	04/06/91	AU	626510 B	30/07/92
_	• • • • • • • • • • • • • • • • • • • •			AU	6922291 A	11/07/91
				CA	2033689 A	11/07/91
				ΈP	0440339 A	07/08/91
			•	JP	6100388 A	12/04/94
				us	4997469 A	05/03/91
				US	5021247 A	04/06/91
JS	5411568	A	02/05/95	NONE		
√O	0121556	A1	29/03/01	AÜ	7293200 A	24/04/01
				FI	992021 A	21/03/01

Information on patent family members

01/10/01

International application No.
PCT/FI 01/00667

	Patent document Publication cited in search report date		Patent family Publication member(s) date			
GB	2112765	A	27/07/83	AT	357382 A	15/10/85
		•••		AT	380469 B	26/05/86
				AU	534003 B	22/12/83
				ĂÜ	8868482 A	31/03/83
			•	BR	8205602 A	30/08/83
				CA	1188124 A	04/06/85
				CA	1197110 A	26/11/85
				CH	657119 A	15/08/86
				CS	259509 B	14/10/88
				CS	8206855 A	15/03/88
				DD	212505 A	15/08/84
				DE	3235523 A,C	14/04/83
				DK	159965 B,C	07/01/91
				DK	424682 A	26/03/83
				ES	515876 A	16/09/83
				ES	522033 A	01/12/84
				ES	8308526 A	01/12/83
				ES	8501727 A	01/03/85
•				FI	72966 B,C	30/04/87
				FI	823273 A	26/03/83
				FR	2513542 A,B	01/04/83
				GR	77675 A	25/09/84 29/03/88
				HU	194794 B	28/03/88 09/11/88
				IE	53417 B 158473 A	22/11/86
				IN IT	158473 A 1157319 B	11/02/87
				JP	1346724 C	13/11/86
				JP	58069795 A	26/04/83
				JP	61012878 B	10/04/86
				KR	8600726 B	14/06/86
				MX	160111 A	30/11/89
				NL	183512 B,C	16/06/88
				NL	8203699 A	18/04/83
				PH	20156 A	09/10/86
				PL	238341 A	23/05/83
				PT	75610 A,B	01/10/82
				RO	85788 A	25/11/84
				SE	448722 B,C	16/03/87
				SE	8205454 A	23/09/82
				US	4554003 A	19/11/85
				US	4559075 A	17/12/85
				US	5139555 A	18/08/92
				YU	43094 B	28/02/89
				YU	212182 A	31/10/85
				BE	894470 A	17/01/83
				US	4560400 A	24/12/85
				US	4832728 A	23/05/89
				ZA	8206609 A	27/07/83
				CA	1188536 A	11/06/85 10/01/86
				PH	19144 A 4421545 A	20/12/83
				US		LV/ 12/ 0J
					V (•